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FINAL REPORT

AEC RESEARCH AND DEVELOPMENT REPORT

Carbide and Carbon Chemicals Corporation Operating Contractor for the U.S. Atomic Energy Commission.

PART III

Research Summary - K-25

(February 16, 1945 - September 30, 1945)

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Submitted in Accordance with
Terms of Contract W-7401-eng-43
(April 1, 1943 - September 30, 1945)

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The Final Report Consists Of:

- Part I - Research Summary - MSA - (4/1/43 to 2/15/45)
Submitted July 3, 1945
- Part I Appendix - Production Summary - MSA - (4/1/45 to 2/15/45)
Submitted Jan. 31, 1946
- Part II - Research Summary - Y-12 - (1/1/45 to 9/30/45)
Submitted Jan. 31, 1946
- Part III - Research Summary - K-25 - (2/16/45 to 9/30/45)
Submitted Jan. 31, 1946

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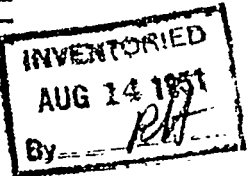
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I. INTRODUCTION.

From April 1, 1943, to September 30, 1945, this laboratory was under contract to the Manhattan Engineer District for both research and production. This Contract, W-7401-eng-43, was, in effect, an extension of OERD Contract OEMsr-512 (6-1-42 to 3-31-43) and was administered by Madison Square Area until February 15, 1945. During the early part of the Contract, the facilities and personnel of the laboratory were also engaged on a similar program under NDRC Contract OEMsr-332 (11-1-41 to 6-30-43) which had previously overlapped NDRC Contract OEMsr-271 (10-15-41 to 11-15-42).

Final Reports were submitted for Contract OEMsr-271 on June 14, 1943, Contract OEMsr-332 on June 24, 1944, and Contract OEMsr-512 on December 12, 1944. A research summary for Contract W-7401-eng-43, from April 1, 1943, to February 15, 1945, was submitted to Madison Square Area and has now become Part I of the Final Report. Production of special materials during this period is summarized in an Appendix to Part I, submitted to Manhattan District Office.

At the close of the work for Madison Square Area it was decided to transfer administration of the contract to the Manhattan District Office. Accordingly the personnel working for Madison Square Area on February 14, 1945, were transferred to work on problems of interest to K-25. The program included research on C-216, fluorocarbons, and handling of enriched T. The present report, Part III of the Final Report, describes the work done by this group from February 15, 1945, until the expiration of the contract September 30, 1945.

Part of the force working for Madison Square Area had already been placed under District Office administration on January 1, 1945, to work on problems of interest to Y-12. This program also continued until the expiration of the contract, September 30, 1945, and is described in Part II of the Final Report.

A complete list of technical reports relating to Part III is attached, and appropriate references are made at the beginning of each section. The numbering system of these reports is a continuation of that begun in Part I and is also keyed to the list attached to Part II.

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II. ABSTRACT.A. Stability of Pump Oils (2/15/45 - 7/31/45).

1. Introduction.
Summary of earlier work leading to the Ignition Temperature Test.
2. Stabilization.
Study of C-216 and C-616 treatment of C-2144 to reduce the latter's reactivity toward these gasses.
3. The Instantaneous Absorption Method.
Development and evaluation of this stability test, and comparison with other tests.
4. Analytical Methods.
Analytical procedures for C-216 gas mixtures and for hydrogen and free acid in C-2144.

B. Recovery of T from a C-216 Disposal System (6/1/45 - 8/30/45).

1. Introduction.
Previous research and preliminary studies of the system.
2. Procedures Involving the T-Peroxy Complex.
Attempts to obtain either immediate and complete precipitation or continuous and complete solution of T in this system.
3. Procedures Involving the T-Carbonate Complex.
Study of techniques for complete and continuous solution in a carbonate system, either with or without continuous fluoride ion removal.

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III. RESEARCH SUMMARY.

A. Stability of Pump Oils.

(References: 30-48, 107, 138-140, 145-151, 153-154, 160, 199-203)

1. Introduction.

The unsatisfactory performance of pumps handling C-216 alone or in combination at site had already led to a re-examination of the two most satisfactory pump fluids; C-2144 and MFL. Although both were being produced under rigid specifications, there was still considerable destruction of the oil, sludging in the pumps, and reaction of the gasses with the fluid. There was sufficient irregularity in the performance of various lots of both of these materials to warrant an investigation of their reaction with C-216, and later with C-616. The early phases of this work are reported in Part I of this Final Report in sections III-10 and III-11.

Since one of the earliest needs was a satisfactory laboratory test which would permit comparison of reactivities toward C-216 (or C-616) the earliest studies had centered on the C-216 reaction, and a test known as the Ignition Temperature had been developed. This consisted of slowly warming a standard sample of oil while a jet of 20% C-216 was bubbled in below the surface. The temperature at which a visible flame occurred at the jet was referred to as the Ignition Temperature or I.T.

The experiments described in Part I of this Final Report were concluded early in the period covered by the present part. The effects of impurities were determined and runs were made with the oil in the form of a mist or vapor and as a thin film on a revolving disk. It was found possible to relate the Ignition Temperature to the percent hydrogen by weight (as determined by pyrolytic analysis) and this test served as the first means of determining the relative reactivities of various specification-grade samples of both types of pump oil. Rather wide differences were found on passing from one lot to another.

Due to the simplicity of the method as well as the apparent direct relationship between I.T. and percent hydrogen this test was adopted as a control in the manufacturing process

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for C-2144. As a further check, this laboratory ran control I.T. determinations on all lots of C-2144 sent from the manufacturer to site. It was at this time believed that unreacted hydrogen was the principal cause of difficulty, and the specification called for an Ignition Temperature above 300° C., corresponding to less than 0.07% hydrogen.

2. Stabilization.

a. Preliminary Observations.

During the course of the above work it had been noted that exposure to C-216 during an I.T. run tended to raise the Ignition Temperature of the oil, suggesting that a portion of the hydrogen was being replaced without destruction of the molecule. Treatment of C-2144 without ignition appeared to have the same effect, and it was possible to prepare a treated C-2144 which would not ignite up to its boiling point (about 330°). This observation led to further investigation in the hope that such treated oil might be more stable in a pump.

b. Stabilization with C-216.

The experimental treatments involved samples of oil up to 3000 ml and C-216 concentrations up to 40% by volume. The effect of C-216 flow rate and concentration, temperature, duration of treatment, degree of agitation, catalyst (FX-30) as well as other variables was studied. The data obtained showed that stabilization toward dilute (20%) C-216 could be achieved with yields greater than 90%. It was also shown that low-hydrogen vapor-phase C-2144 stabilized much faster than liquid-phase material.

The degree of stabilization was found to be primarily temperature dependent. The C-216 flow rate, concentration, and degree of mixing, were found to affect the rate rather than the degree of stabilization. Optimum conditions, as found here, were the use of 20% C-216 at a gas flow rate of 3 cc/cc of C-2144, with agitation being limited only by losses due to entrainment in the gas stream. The use of FX-30 as a catalyst appeared to improve the yield, although this was not conclusive. Results were most satisfactory when the starting temperature was such as to give 100% absorption of inlet C-216, and the temperature gradually increased to maintain such absorption until an arbitrary termination temperature (usually 300° C.) was reached. This temperature was then maintained until C-216 absorption fell to a low and constant value.

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The stability of MFL was found to lie between treated and untreated C-2144, and attempts to stabilize it by the above technique were unsuccessful. Considerable cracking took place and the end product was still not as stable to C-216 as treated C-2144.

c. Stabilization with C-616.

Samples of C-216 treated and untreated C-2144 were exposed to C-616 under conditions duplicating those of a stabilization run. Measuring the C-616 absorption by the amount of non-volatile T found in the sample, very little stabilization took place with the treated C-2144. With the untreated material there was a two-fold reduction in the reactivity as a result of C-616 treatment. The reduction in reactivity was three-fold in the case of MFL.

The reactivity values referred to above, obtained using C-616, also permitted a comparison of the C-616 stabilities of the three liquids. As was expected the treated C-2144 was more stable than untreated toward C-616 by a factor of four. MFL, however, which, based on C-216 stabilities, had been intermediate between the two types of C-2144 was found to be more reactive towards C-616 than the untreated C-2144, by a factor of seven. C-616, therefore, as a means of measuring stability, parallels C-216 only for C-2144 samples.

d. A Test Sample of Stabilized C-2144.

It had been found that by use of the stabilization technique described in section b. it was possible to reduce the reactivity of C-2144 to such an extent that only 3% of C-216 fed at 300° C. was absorbed. This may be compared to 100% absorption at about 200° C. using untreated oil. A three-gallon sample for test in operations at site was prepared in a semi-pilot scale reactor. The average yield was 88%, and the oil had an absorption of 8% at 300° C. Further supplies of such material were produced as requested, but due to operational difficulties the results of the tests were meaningless.

3. The Instantaneous Absorption Method.

a. Technique.

With the increased stability being achieved as a result of C-216 treatment, the Ignition Temperature was no longer adequate to give an indication of quality. The test had likewise become useless in connection with production lots which were being after-treated by other means to improve

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their stability. Other analytical methods also being uncertain, efforts were made to find a new procedure to distinguish quality in these treated samples.

It had been noted that as a treatment run progressed, absorption of C-216 by the liquid dropped slowly. This was investigated to see whether under standard conditions the percent absorption would be a measure of the quality of the C-2144. Since stabilization of the material undergoing test would occur continuously it was necessary to make the test of short duration, and it was found that if the C-216 were sampled within five minutes after the gas was admitted results were reproducible. The amount of absorption or Instantaneous Absorption at the reference temperature was designated as the I.A. value. Customarily C-216 was passed through liquid C-2144 at 300° C. for stabilized material and at lower temperatures for all other samples.

b. Study of Variables.

A series of measurements were made on standard samples of C-2144 to determine the effects of variation in flow rate and concentration of C-216 from the standard of 100 cc 20% C-216 per minute through a 100 ml sample of C-2144. The variations were found to be small. The reproducibility of the method is good provided that contamination of the system is carefully avoided. Blank tests of the order of 1% C-216 absorption can be obtained, and the method will give checks in I.A. values to within one or two units.

c. The I.A. Values for Various Oils.

After standardizing the method, a series of measurements were made on various samples of treated and untreated C-2144 and MFL to determine the range of applicability of the test. With treated C-2144 of high stability the I.A. value was the only property found to be directly related to the duration and temperature of C-216 treatment.

I.A. measurements on untreated C-2144 were in general not satisfactory. Samples which had an Ignition Temperature, and even some which had been treated beyond this point, all showed values in the range of 55-65% at 150° C. At temperatures above this the absorption was uniformly 100%, though the Ignition Temperatures and previous histories of these samples were widely different. Samples included both vapor- and liquid-phase C-2144, extracted vapor-phase C-2144 and others. With MFL the I.A. method was of very little use, the best sample having an I.A. of 75% at 300° C. while untreated material fell in the same range as untreated C-2144.

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To make a final check on conditions to be expected in the plant, I.A. determinations were made using mixtures of C-216 and C-616. The two compositions chosen were 20% and 40% C-216, each with 5% C-616. A series of measurements at 250° C. on several C-2144 samples indicated absorptions of each component as would have been predicted had they been used separately.

d. Comparison of Stability Tests.

During 1945, there were in use four different tests or analyses for predicting the stability of pump oils. The Ignition Temperature and the percent hydrogen by weight showed a consistent relationship which was useful over the range from approximately 0.40-0.07%, but in this same range the I.A. test was unable to distinguish between samples. In the range where the I.A. method is applicable the analysis for hydrogen becomes a matter of great experimental difficulty and uncertainty, and no direct relationship between the percent hydrogen and the I.A. could be established. Based on the best data available, it is believed that stabilized C-2144 having an I.A. of about 5% at 300° C. had a hydrogen content of 0.005%. The fourth method, the Inertness Residue Test involved measurement of the extent of reaction with C-616 and was not successfully related to the I.A. or I.T. value.

The conclusion was reached that stability under operating conditions was not a simple question of hydrogen content and that possibly none of the four test methods available would be satisfactory. Further studies were planned, based on investigation of molecular configuration, bond unsaturation, etc., but were deferred since production was temporarily suspended and the material on hand was performing relatively well.

4. Analytical Methods.

a. C-216 Analysis in Gas Mixtures.

For purposes of routine work, the Columbia method of analysis, based upon absorption of C-216 by mercury was too time-consuming, since the absorption and subsequent gas analysis took about twenty minutes per sample. The use of a KI solution to absorb the C-216, followed by titration for liberated I₂ was therefore investigated and subsequently adopted, since the time required was as little as three minutes per analysis. In this method, the C-216 mixture to be analyzed was sampled at a slight positive pressure in a calibrated gas sampling tube

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(EO-100 ml). A ten percent KI solution was then admitted to the bulb, the C-216 absorbed, and the I_2 titrated with $K_2S_2O_8$. This method was checked against the mercury method over a C-216 concentration range of 5-40% with excellent agreement, and was successfully used to analyze exhaust C-216 from C-2144 treatments. For both methods, it was desirable to use a cold trap ahead of the sampling system to avoid contamination by C-2144.

To avoid intermittent sampling, a continuous, colorimetric method of analysis, devised by Columbia and Kellex, was studied. This method, based upon the displacement of Br_2 gas from KBr by C-216, was extended to cover a range of C-216 concentration up to 50%. The entire Br_2 system, including the absorption cell, was thermostated at an elevated temperature to prevent condensation. Agreement with other methods was excellent, and the technique was used to follow exhaust C-216 concentration during stabilization runs on C-2144.

b. The Determination of Hydrogen in C-2144.

When the possibility of using the fluorescence of C-2144 to measure its hydrogen content was reported, measurements were made to test this method for possible use in stabilization work. While the fluorescence of production lots of C-2144 was found proportional to their hydrogen content, no such relationship was found in samples which had been treated with C-216. The fluorescence appeared to be chiefly a function of color and not hydrogen content and the results were anomalous with samples which had become colorless through treatment with C-216. This was confirmed by experiments on distillation fractions of untreated C-2144 which also gave anomalous results. The color tended to concentrate in the residue, resulting in high fluorescence though the hydrogen content of these residues was normal.

The pyrometric method of analysis for hydrogen in C-2144 was tried in this laboratory, and while useful for material having larger amounts of hydrogen (0.1-0.5%), was found unsuitable in the lower range (less than 0.07%). A standardization program was attempted, but experimental difficulties such as inconsistent and high blanks, non-reproducibility, and short equipment life, prevented a satisfactory conclusion.

c. An Investigation of Acid in C-2144.

A few preliminary measurements were made on samples of C-2144 to determine the amount of free acid. These measurements consisted of titration for acid in water used to

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extract C-2144. The aqueous layer was replaced after each analysis, and a rapid decrease to a constant value suggested both free HF and HF from hydrolysis. Apparent HF contamination of the order of 0.001% was found.

B. Recovery of T from a C-216 Disposal System at K-28.

(References: 202-205)

1. Introduction.

During an operation at site, small quantities of C-616 were discharged to a caustic disposal system designed to handle C-216. In this system, the gases were absorbed in a carbon-lined scrub tower using a 10% caustic solution, fluoride ion being removed and the caustic solution continuously regenerated by addition of $\text{Ca}(\text{OH})_2$. Under these conditions, it would be expected that any T reaching the tower as C-616 would be absorbed and the T precipitated in the form of a very insoluble dituballate of either sodium or calcium. However, it was found that the solution was yellow-brown after a period of several weeks and upon analysis, a soluble T compound was found to be present.

Preliminary research by Kellex had shown that solution would occur only when C-616 was passed into caustic with or followed by C-216. The soluble T was therefore thought to be in the form of a pertuballate; the peroxide having resulted from C-216 absorption. T could be precipitated from this complex slowly by addition of $\text{Ca}(\text{OH})_2$ and rapidly by addition of $\text{Ba}(\text{OH})_2$.

This laboratory was asked to undertake a study of the safe-handling and recovery of all T reaching the disposal tower. The danger of accumulations of T at any point in the system made it imperative that it be handled only under very close control, and since the presence of T in solution appeared due to formation of the pertuballate complex, preliminary experiments were carried out on the solution chemistry of this compound.

It was shown that a stable complex can be formed by addition of peroxide to caustic containing dituballate, or by addition of T-peroxide (TO_4) to caustic, or by absorption of C-216 - C-616 mixtures. The approximate solubility was measured in 5, 10, and 20% sodium hydroxide and found to be 0.007 M, 0.02 M, and 0.10 M in T, respectively. A ten-fold excess of peroxide was found to decrease the solubility and to increase the tendency toward supersaturation.

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The peroxy-tuballoy system is apparently complex and the pertuballate does not appear to have a unique formula in solution.

2. Procedures Involving the T-Peroxy Complex.

a. Immediate Removal by Precipitation.

According to Kellex data, about 10% of the T reaching the tower as C-616 went into solution. The remainder precipitated immediately as a dituballate, and could, therefore, be readily filtered off at the base of the tower. That which went into solution was very slowly precipitated by $\text{Ca}(\text{OH})_2$ in the lime pit and was therefore mixed with CaF_2 and excess lime. Recovery from the lime was difficult and the accumulation was uncontrollable.

In an effort to prevent the formation of the soluble complex, and to destroy peroxide formed by C-216, a variety of reducing agents were added to the tower solution, and, although they improved the C-216 absorption, they did not successfully destroy the soluble T-complex; such compounds included Na_2S , ClO^- , SO_3^{2-} , and $\text{S}_2\text{O}_3^{2-}$.

Since it did not seem possible to prevent the formation of the soluble T-complex in caustic, other precipitation agents were investigated. It was found that barium in the form of solid $\text{Ba}(\text{OH})_2$ was from 10 to 100 times more efficient for the purpose than calcium. No other ion was found which was both relatively cheap and efficient in removing T from an alkaline solution of pertuballate. It was hoped that barium might be used to remove T, after which fluoride removal would be attempted by means of the lime pit in the ordinary manner.

For this to be successful it would be necessary that the solubility of calcium fluoride be much lower than that of barium fluoride, both to avoid contamination of the barium reactor with large amounts of fluoride and to limit the amount of barium hydroxide required. This was not true in 10% caustic but measurements showed that if the caustic concentration were lowered, barium fluoride became relatively more soluble. A compromise was selected at 5% caustic where there is almost a 3-fold difference in the two solubilities.

It was further recognized that under the varying conditions of operation the composition of the resulting solid barium salts in the T-removal stage would vary, including more or

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less BaF_2 and BaCO_3 , the latter arising as a result of contamination from air. It was accordingly necessary to determine the effect of the latter compounds of barium on the precipitation of T. They were measured and it was found that BaF_2 was very nearly as good as $\text{Ba}(\text{OH})_2$, but that BaCO_3 was very poor. All three, however, were much more efficient than the corresponding calcium salts.

A complete system was tested on an experimental tower, adding excess $\text{Ba}(\text{OH})_2$ between the tower exit and the lime pit and allowing the slurry to settle. The clear solution then passed through a lime pit and into a second settling tank where the fluoride was removed. Solid barium salts of T were formed as predicted, and the T concentration in the effluent from the barium settling pit was satisfactorily low.

Using 5% caustic, the efficiency of the Tower for C-216 absorption was somewhat decreased. This reduction amounted to less than 10% and should not be serious in practice. The presence of CO_3^{2-} was not troublesome in the laboratory experiment, but if any considerable quantity of barium were to exist in this form, the efficiency for removing T was shown to be dangerously impaired.

Based on these data, a procedure was suggested to Kellogg which would permit precipitation and recovery of all T from caustic tower effluents. This was the same as the successful laboratory technique with addition of a carbonate-removal section preceding the scrub tower.

b. Continuous and Complete Solution.

During the early part of July, 1946, the emphasis of work was shifted by the Kellogg Corporation to the complete solution of T, since the precipitation method was limited to unenriched material. The addition of peroxide was suggested as a means of holding all the T in solution. This would not involve any change in the existing system and would eliminate the possibility of precipitating dangerous amounts of enriched T due to surges.

By operating an experimental tower to which peroxide was continuously added, it was shown that C-616 and mixtures of this gas with C-216 were absorbed completely without any precipitation. The C-616 concentration was varied from the highest anticipated value of 5% to 0.05%.

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For safe operation, it would have been necessary to maintain a high peroxide concentration at all times, even though C-616 reached the tower only occasionally. Because of the instability of the peroxide in the existing caustic solution, continual maintenance of this high concentration would have been prohibitively costly. Accordingly, a study was made of this instability, not only from the standpoint of materials of construction and solution impurities, but also to determine whether there were any practical stabilizing agents.

It was found that silver solder, carbon, steel and glass, all of which were in use in the system as it was then operating, had an accelerating effect on the rate of peroxide destruction. Silver solder, used extensively in piping and gas lines at site, was particularly bad. A number of stabilizing agents, suggested by a literature search, were tried, best results being achieved with MgO , $MgSO_4$ and $Mg_3(PO_4)_2$. With the latter agent, in the absence of metals or other impurities, considerable stabilization was achieved, but in the presence of metals, the stabilizing effect was not sufficient.

A further problem consisted of the precipitation of T in the lime pit used for fluoride removal. It was hoped that the presence of excess peroxide might prevent this, but in the laboratory it was shown to have the opposite effect, a very slight excess greatly increasing the extent of T precipitation. A quantitative study was also made of the precipitation by $Ca(OH)_2$ in the absence of excess peroxide and it was found that amounts in excess of those fed to the tower at site caused almost complete precipitation, while amounts equivalent to or less than those used in operation (0.5 gm/L) were sufficient to precipitate traces of T from synthetic solutions of the complex.

Due to the instability of peroxide and the coprecipitation of T in the lime pit, it was not possible to recommend the peroxide addition method for use in the existing system.

3. Procedures Involving the T-Carbonate Complex.

a. Continuous and Complete Solution without Fluoride Removal.

With the failure of the peroxide stabilization investigation, the T-carbonate complex was regarded as the next most likely means of retaining T in solution. Preliminary

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solubility data, obtained in other laboratories, indicated that a 5% Na_2CO_3 solution would dissolve sufficient T for practical operation, although this would require rather extensive changes in the existing equipment, since lime could no longer be used to remove fluoride ion.

It was suggested that batchwise operation be resorted to, in which the carbonate solution would be allowed to absorb both T and fluoride until nearly saturated in respect to the latter. The entire solution would then be removed, acidified to destroy CO_3 , and any one of a number of procedures used to recover T. The solubilities were such that saturation with respect to T, deriving from C-616, would not occur before saturation with respect to fluoride.

A major problem in the use of this solution was the relatively inefficient absorption of C-216. Upon investigation, it was found that if the efficiency of 10% NaOH be assumed to be 100 the efficiency of 5% carbonate would be approximately 75. It was, however, found that the addition of certain reducing agents (0.01 M $\text{Na}_2\text{S}_2\text{O}_3$ or $\text{Na}_2\text{S}_2\text{O}_5$) increased the efficiency to that of 10% caustic, and had no measureable effect on the stability of the T-complex.

The system was put in operation in an experimental tower, and no difficulties were experienced in retaining T in solution up to the point of saturation with respect to fluoride. Three methods of T recovery were investigated and found to work satisfactorily, although much of this work was duplication of intensive studies elsewhere. The fluoride solubility was found to be about 2.7 gm/100 ml, which would give an estimated operating period of several months.

During operation it was found that the pH fell, and the carbonate was gradually changed to bicarbonate, as a result of the acid liberated when the gases were absorbed. This had only a slight effect on the efficiency of C-216 absorption, and did not interfere at all in C-616 absorption; the complex being more soluble in bicarbonate than in carbonate. However, from an operating point of view, the circulation of bicarbonate solutions was not practical due to the materials of construction, and it was necessary to determine whether the pH could be kept constant by the addition of controlled amounts of caustic. It was found that if high local caustic concentrations were avoided, the pH could be maintained. Using this technique, concentrations of soluble T as high as 10,000 mg/L were achieved at pH 9.8-10.0. On the basis of these data, a final batchwise procedure was recommended to Kellogg.

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b. Continuous and Complete Solution with Fluoride Removal.

The disadvantages of the above batchwise process were sufficiently serious to justify further research toward making it continuous. Control of pH was more difficult than in the caustic system, and periodic handling of the entire volume of carbonate solution was inconvenient. To correct this, it would be necessary to provide for the removal of fluoride without precipitation of T, and to maintain a constant pH, as is done in the caustic system by the use of lime. An investigation was therefore undertaken to determine if either or both of these objectives might be attained.

From solubility data, it was possible that either CaCO_3 or MgCO_3 might be suitable for fluoride removal. These were investigated and it was shown that both precipitated fluoride slowly from 5% carbonate solution, MgCO_3 being the more rapid. This latter compound appeared to have no effect on the concentration of the T-complex, whereas the calcium was unsatisfactory in this respect.

The use of MgCO_3 , however, did not provide a constant pH, and some attention was devoted to the use of MgO (or Mg(OH)_2) despite the fact that it is almost as insoluble as MgF_2 . Quantitative experiments, at pH in the neighborhood of 11, indicated no precipitation of T from the carbonate complex and satisfactory fluoride removal. However, when acid was added in an amount roughly corresponding to the fluoride present, 80% of the carbonate was converted to bicarbonate, and upon addition of MgO , both T and fluoride were rapidly precipitated.

In a similar test using a small tower, however, where the pH was about 11 at the start, and in which the carbonate solution subsequently passed through a slurry pit containing MgO , the concentration of T built up to almost 12,000 mg/L without apparent precipitation. The pH remained constant at 10, while the fluoride concentration was maintained at 1300 mg/L compared to a calculated 12,000 mg/L if no precipitation had occurred. The wet solids in the slurry pit were light yellow and contained at least some T. A portion of the color could be washed out with water, and it was not certain whether the color remaining was due to insoluble T-compounds or contamination from the solution.

Although this investigation was incomplete, experimental work was terminated due to contract expiration, and the final evaluation of the MgO process was carried forward by another laboratory. No recommendation could be made by this laboratory on the basis of available data.

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V. LIST OF RELATED REPORTS.

The following list supplements those found in Parts I and II of the Final Report, and together with them gives a complete record of technical reports prepared under Contract W-7401-eng-43. Also attached is an addendum consisting of reports previously listed in Part I and referred to in Part III. The numbers are used as references in the text of Part III of the Final Report.

I. ROUTINE REPORTS.

Monthly Progress Reports

- | | |
|---------------------|------------------|
| 192. March 1945 | 195. June 1945 |
| 193. April 1945 | 196. July 1945 |
| 194. May 1945 | 197. August 1945 |
| 198. September 1945 | |

Monthly Technical Reports

- | | |
|---------------------------------------|----------------------------|
| 199. Feb. 15 - Mar. 31, 1945 (M-2123) | 202. June 1945 (M-2129)✓ |
| 200. April 1945 (M-2126) | 203. July 1945 (M-2131) - |
| 201. May 1945 (M-2127) | 204. August 1945 (M-2133)✓ |
| 205. September 1945 (M-2135)✓ | |

II. GENERAL REPORTS.

160. Hydrogen Analysis of C-2144 by Ignition Temperature.
(Feb. 17, 1945)
161. Stability of H_3PO_4 with Fluorine.
162. Final Report - Part I - July 3, 1945
163. Part I - Production Appendix - Jan. 31, 1946
164. Part II - Jan. 31, 1946
165. Part III - Jan. 31, 1946

III. LETTER REPORTS.

134. Molecular Weight Data on C-716 Distillation Cuts.
(Mr. F. B. Downing - June 18, 1943)
135. Molecular Weights of Fluorocarbons by the Freezing Point
Technique. (Mr. F. B. Downing - Nov. 18, 1943)
136. Vapor Pressure of a Composite of GD 1180 and GD 1181.
(Dr. R. Rosen - July 12, 1944)
137. Preparation of High Boiling Fluorocarbons.
(Dr. R. Rosen - Sept. 22, 1944)

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138. Reactivity of C-2144 to C-216.
(Capt. W. M. Hearon - Aug. 28, 1944)
139. Standardization of Pyrolytic Method for C-2144 Analysis.
(Capt. W. M. Hearon - June 25, 1945)
140. Reactions of C-2144 with C-216.
(Maj. A. F. Thompson - Nov. 10, 1944)
141. Purification Methods for C-2144.
(Maj. A. F. Thompson - Nov. 29, 1944)
142. Use of FM-44 in Preparation of C-2144.
(Capt. W. M. Hearon - May 26, 1944)
143. Data on Samples of Extracted C-2144.
(Capt. W. M. Hearon - Mar. 7, 1945)
144. Preparation of C-2144 Residue.
(Capt. W. M. Hearon - Jan. 18, 1945)
145. Data on Samples of Stabilized C-2144.
(Capt. W. M. Hearon - Feb. 28, 1945)
146. Evaluation of Pyrolytic Method of Analysis for C-2144.
(Dr. C. F. Irwin - July 19, 1945)
- Data on Samples of Stabilized C-2144.
147. (Mr. H. F. Priest - Mar. 19, 1945
148. May 22, 1945
149. June 5, 1945)
- Comparison of Stability Tests for C-2144.
150. (Mr. H. F. Priest - April 21, 1945
151. June 20, 1945)
152. Physical Properties of Allis Chalmers Sample NC-716.
(Dr. R. Rosen - June 24, 1944)
- Data on Samples of Stabilized C-2144.
153. (Dr. R. Rosen - Dec. 12, 1944
154. May 16, 1945)
155. Properties of C-2144 Prepared with FM-66.
(Dr. R. Rosen - June 21, 1944)
156. Extraction Purification of Fluorocarbons.
(Lt. Col. J. R. Ruhoff - Mar. 28, 1944)

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- 157. Pressure-Stats on C-216 Lines.
(Dr. R. Rosen - Dec. 20, 1943)
- 158. Physical Properties of Certain Fluorocarbons.
(Dr. R. Rosen - Sept. 14, 1943)
- 159. Properties of C-2144 Residues.
(Dr. R. Rosen - Sept. 14, 1943)

ADDENDUM

(Reports Previously Listed in Part I)

Monthly Technical Reports

- | | |
|--------------------|----------------------------|
| 30. September 1944 | 33. December 1944 |
| 31. October 1944 | 34. January 1945 |
| 32. November 1944 | 35. Feb. 1 - Feb. 15, 1945 |

Weekly and Bi-Weekly Technical Reports

- | | |
|-----------------------------|-----------------------------|
| 36. Oct. 30 - Nov. 5, 1944 | 42. Dec. 11 - Dec. 16, 1944 |
| 37. Nov. 6 - Nov. 12, 1944 | 43. Dec. 18 - Dec. 23, 1944 |
| 38. Nov. 13 - Nov. 19, 1944 | 44. Dec. 25 - Dec. 30, 1944 |
| 39. Nov. 20 - Nov. 26, 1944 | 45. Jan. 1 - Jan. 6, 1945 |
| 40. Nov. 27 - Dec. 3, 1944 | 46. Jan. 8 - Jan. 13, 1945 |
| 41. Dec. 4 - Dec. 10, 1944 | 47. Jan. 15 - Jan. 27, 1945 |
| 48. Jan. 29 - Feb. 15, 1945 | |

Letter Reports

- 107. Treatment of C-2144 with C-216.
(Capt. W. M. Hearn - Feb. 6, 1945)

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V. BUDGET.

The following figures represent expenditures through September 30, 1945, plus outstanding obligations and credits due as of this date. A duplicate table is attached to Part II.

Salaries	\$ 362,787.51
Equipment & Supplies	258,606.07
Subcontracts	5,203.00
Overhead	<u>181,393.76</u>
Total	\$ 807,990.34

This does not include the costs of contract termination proceedings and building restoration, nor credits resulting from the sale or return of property after September 30, 1945.

For The Johns Hopkins University

W. B. Burford, III

W. B. Burford, III

APPROVED BY:

D. C. Moore
D. C. MOORE,
Major, Corps of Engineers.
AUTHORIZED REPRESENTATIVE OF
THE CONTRACTING OFFICER.

APPROVED

N. K. Archer
N. K. ARCHER
MAJOR, C. E.

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